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U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

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U.S. DEPARTMENT OF COMMERCE
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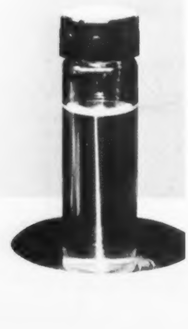

COVER

A low-power laser illuminates traces of dust in a chemical reagent. An NBS technique uses the amount of light scattered by the laser to indicate the contamination level of the liquid. This vial shows a particle contamination of approximately 0.2 ppm. (See page 80.)

Prepared by the NBS Office of Technical Information and Publications, Washington, D.C. 20234
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The National Bureau of Standards serves as a focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. For this purpose, the Bureau is organized into three institutes—

- The Institute for Basic Standards
- The Institute for Materials Research
- The Institute for Applied Technology

The TECHNICAL NEWS BULLETIN is published to keep science and industry informed regarding the technical programs, accomplishments, and activities of all three institutes.

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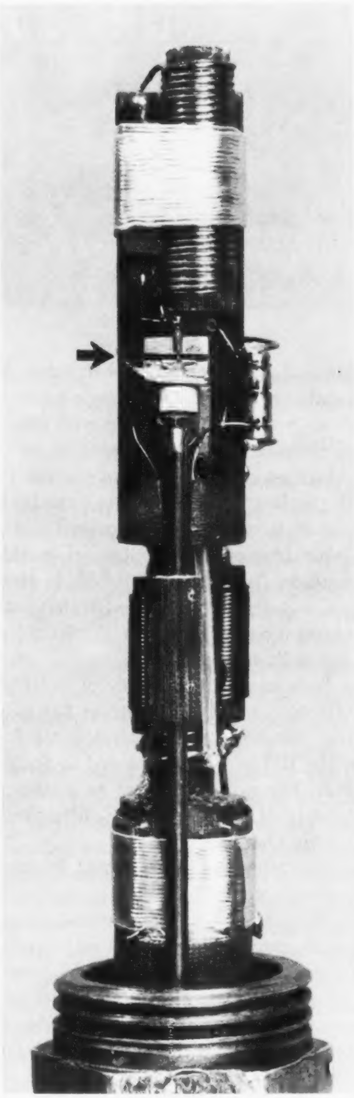


The arrangement of wires on the rectangular metal block is the adjustable Josephson junction (arrow); voltage bias and thermal noise is supplied by the resistor (right).

NBS ADVANCES NEW CONCEPT IN LOW-TEMPERATURE MEASUREMENT

R. A. Kamper makes a final adjustment of the Josephson junction before inserting it into a cryostat.

■ In the temperature range below 10 K, and especially below 1 K, thermometry becomes very difficult. The need for temperature measurements at these low temperatures, however, has been steadily increasing. This need is especially felt in studies of rocket fuels, superconductivity, nuclear magnetism, and many other areas of science and technology.

Since traditional methods of temperature measurement become difficult to use in the millidegree range, more exotic concepts have begun to appear more practical. One such possibility was recently advanced by R. A. Kamper of the NBS Institute for Materials Research at the Cryogenics Laboratories, Boulder, Colo.[1] This concept offers the promise of a thermometer which can measure directly the absolute temperature of a metallic conductor in the temperature range from 0.001 to 10 K. An important advantage of the thermometer, in principle at least, is that no calibration is required.

The phenomenon on which the prediction of temperature measurement is based is electrical noise—a fluctuating voltage which is present in all metallic conductors due to the Brownian motion of the electrons. The magnitude of this voltage varies as a function of

continued

both temperature and resistance of the conductor and becomes very small at low temperatures. However, it should be detectable at very low temperatures with the aid of the Josephson effect, which occurs when two superconductors are connected by a weak junction. When a steady bias voltage is maintained between the two superconductors, the junction oscillates at a frequency proportional to the voltage.

The required bias voltage may be conveniently supplied by passing direct current through a resistor connected in parallel with the junction. The electrical noise in this resistor then causes a random frequency modulation of the signal from the junction, spreading it over a band of frequencies.

A preliminary calculation shows that the width of this band of frequencies is proportional to the value of the resistance and its absolute temperature. Thus, in theory, measuring this bandwidth forms the basis of a new type thermometer.

Preliminary experimentation by A. H. Silver and J. E. Zimmerman, of the Ford Scientific Laboratory, and R. A. Kamper,[2] of the Bureau staff, has confirmed the predictions of the calculation over a wide range of resistances and a modest range of temperatures. Although the thermometer has not yet been tested at millidegree temperatures, realistic values for the bias resistance and system stability indicate the possibility of measuring temperatures in this range. To do this, however, it will be necessary to eliminate all spurious noise associated with the junction. It is apparent, therefore, that some development remains on the thermometer. This further development is currently in progress.

[1] **Millidegree noise thermometry,** by R. A. Kamper, Proc. Symp. Physics of Superconducting Devices (University of Virginia, Charlottesville, Apr. 28–29, 1967).

[2] **Contribution of thermal noise to the linewidth of Josephson radiation from superconducting point contacts,** by A. H. Silver, J. E. Zimmerman, and R. A. Kamper, Appl. Phys. Letters **11**, 209 (1967).

PRECISE TEMPERATURE CONTROL

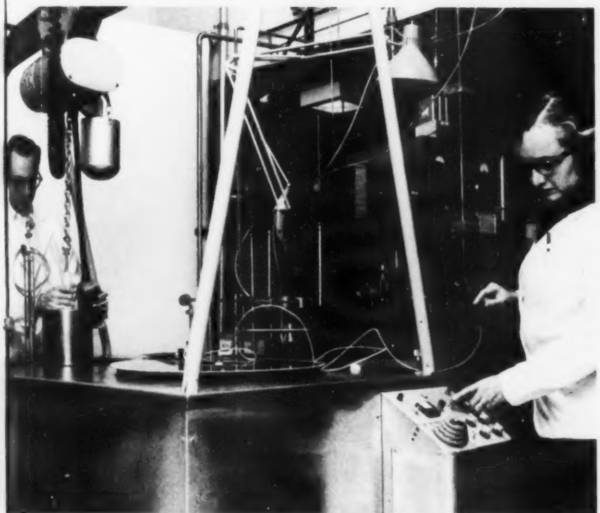
Water Bath Stable to 25 Millionths of a Degree

■ Physical and chemical measurements that must be made in temperature-stable environments include molecular weight determinations, voltage comparisons with standard cells, and energy measurements by several types of calorimeters. Microcalorimeters (devices for measuring minute heat changes) used at the NBS Radio Standards Laboratory as the national standards of microwave power require especially stable temperature control. For this purpose, two RSL scientists, N. T. Larsen and M. E. Harvey, recently developed a precisely controlled water bath that provides temperatures constant to within 25 millionths of a degree Celsius for a 24-hour period.

Previously a water bath having a stability of ± 0.005 deg C was used at NBS to control temperatures for calibration purposes. This, however, proved inadequate for new power standards. The RSL scientists decided to design an improved water bath for which they set as a design goal a temperature stability of ± 0.0001 deg C, fifty times more stable than the existing bath.

The system design that they finally selected isolates the temperature-controlled volume from major paths of energy exchange. It employs a temperature controller containing an a-c bridge with inductively-coupled ratio arms. All the bridge components that would be affected by temperature are located within the controlled-temperature environment. The temperature stability of ± 0.000025 deg C attained by this system is four times better than even the ambitious design goal.

In the past year many of the NBS microwave power transfer standards were calibrated in the new stable-temperature environment. These transfer standards, in turn, were used to make approximately 1400 calibrations of primary laboratory standards sent to NBS by industrial and military standards laboratories. When returned, these



Fred Clague lowers an 18–26.5 GHz power standard into a water bath stable to 25 millionths of a degree. Anne Rumfelt admits water to a second bath containing another radiofrequency power standard.

laboratory standards are used in many thousands of routine calibrations and tests.

Water Bath

The water bath design accelerates calorimeter equilibration by permitting rapid adjustment of the bath to the calorimeter internal temperature. Since this temperature may be either slightly above or slightly below the laboratory room temperature, the control system must be able either to heat the bath's water or to cool it—pump heat from it—in maintaining the desired temperature. The RSL scientists chose evaporative cooling of water for the latter method because it is comparatively simple, is economical, and has a high coefficient of performance (extracts much heat for the energy used).

An important feature of the water bath is that the temperature-controlled volume is within a baffle tank which is surrounded, for the most part, by water. A propeller circulates water up into the temperature-controlled volume, from which it flows radially outward and then down around the outside of the baffle tank. This places a layer of moving water around most of the controlled volume, where most of the heat exchange takes place.

The bath can be operated, when used with an appropriate temperature controller, at any temperature from 18 to 28 °C, provided this temperature is not more than 6 degrees below ambient room temperature.

Temperature Controller

Bath temperature is controlled by a system composed of a platinum resistance thermometer, an amplifier, and a heater, connected in a feedback loop. To obtain sufficient sensitivity and to avoid thermal emf's, a precision alternating-current bridge is used. This permits the use of inductively coupled ratio arms for two of the bridge arms. Their ratio is continuously variable, so that the remaining resistive arm may be a fixed reference resistor. It is located in the probe with the temperature sensor. The temperature coefficient of the inductively coupled ratio arms is negligibly small, virtually eliminating the effect of ambient temperature fluctuations on the sensing bridge circuitry.

Electrical leads entering the bath are arranged in such a way that temperature-induced resistance changes have negligible effect on the bridge. A high gain, low-noise solid state amplifier drives the silicon-controlled rectifier circuit which powers the temperature control heater.

The temperature sensor is an important feature of the temperature controller. Thermocouples were rejected because of their need for a reference temperature. Temperature measurements made by means of the pyroelectric effect also were rejected, because of inadequate long-term stability. Thermometry using the temperature coefficient of quartz crystal oscillators also was rejected, because of high sensitivity to room temperature variations. The choice of a temperature sensor was thus reduced to the temperature-sensitive resistor types.

Performance

The measurement of the stability of the bath and controller presented serious difficulties to the RSL scientists because the thermal stability of any other known temperature measuring system was inferior to that of the new bath. However, Mr. Harvey devised a thermocouple system to measure temperature fluctuations during periods that were short compared to the thermal time constant of a large reference mass. The system provided useful information on fluctuations occurring during intervals of a few seconds to an hour or so, but did not permit measurement times extending to a day or more. This problem was solved by using a second controller as a thermometer, an approach similar to that used for evaluating the stability of standard cells and frequency standards by intercomparing two or more items and measuring the relative drift. This technique makes it possible to identify an item that varies with respect to the mean of an entire group and thus to draw conclusions about its probable stability.

Tests made over a number of days revealed that the bath temperature did not change by more than ±0.000010 deg C during a 9-hour working day. Thermal gradients in the bath in a 30-liter volume were found to be no greater than 0.000009 deg C for bath temperatures between 1.75 deg C above room temperature and 1 deg C below room temperature.

NEW TOOL FOR ENERGY TRANSFER STUDIES

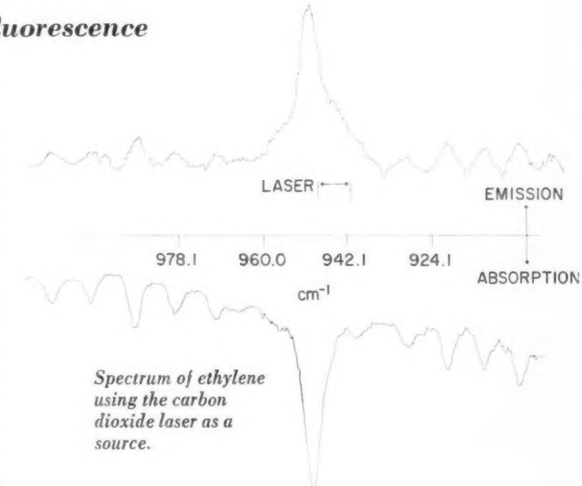
Carbon Dioxide Laser Induces Infrared Fluorescence

■ The recent availability of high-power infrared lasers has stimulated the divergent interests of molecular spectroscopists. In particular, the carbon dioxide laser has been gaining widespread application as an intense source of infrared radiation; its use has been reported in a large number of spectroscopic experiments. Relatively little work, however, has been done on fluorescence using this laser as a source.

In a recent study,[1] by A. M. Ronn of the NBS Institute for Basic Standards, infrared fluorescence experiments were performed on a number of gases by irradiation with a 50-watt continuous-wave carbon dioxide-nitrogen-helium laser. Infrared emission spectra were obtained from a variety of gases. The study, sponsored by the Advanced Research Projects Agency of the Department of Defense under Project Defender, has shown that this experimental procedure is a valuable method of obtaining information for energy transfer studies.

The emission spectrum of ethylene is typical of the results obtained. In this case, a pressure of 50 mm of Hg was found to be optimal for emission with all features of the ν_7 band (approximately 900 to 1000 cm^{-1}) appearing quite well. At that pressure, ethylene absorbed all of the laser lines, and consequently a considerable number of molecules were promoted to the upper state of ν_7. Redistribution of the excited molecules to all rotational states in the vibrational band by thermalizing collisions was very rapid at this pressure, on the order of 0.1–10 microseconds. Vibrational relaxation, however, was much slower, approximately 0.1–10 milliseconds; hence, emission occurred in most rotational transitions in the band.

Similar experiments were performed on sulfur hexafluoride, ammonia, methyl bromide, deuteromethyl iodide, deuterium sulfide, methylamine, methyl acetyline, and ethyl chloride, all of which exhibited transitions in the neighborhood of the laser's frequency. All but deuterium sulfide and methylamine showed fluorescence in varying intensities and at various optimal pressures. The only noteworthy exception appeared in the emission spectrum of ammonia.



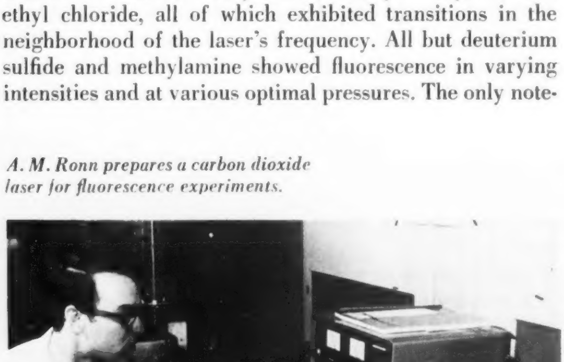
Spectrum of ethylene using the carbon dioxide laser as a source.

In NH_3, coincidence with the laser's lines appeared to be mainly in the overtone band $2\nu_2^a \leftarrow \nu_2^s$, suggesting that the resulting emission spectrum should be distinctly different from the absorption spectrum where $0^a \rightarrow \nu_2^s$ and $0^s \rightarrow \nu_2^a$ lines prevail. This was, indeed, found to be the case as all emission lines originating from the s states were slightly more intense than those from the a states. The overtone transitions for NH_3 were also more intense in the emission spectrum in proportion to the fundamenal Q-branch (band center) transitions than they were in absorption.

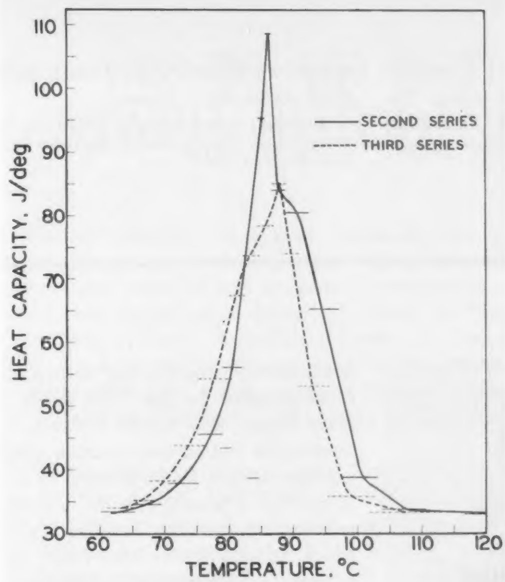
A commercial CO_2 laser was used in the NBS study as a pumping source and an infrared grating spectrometer for detection (resolution 0.5 cm^{-1}). The specimen chamber had four sodium chloride windows on a 25-mm bore "cross" of approximately 10 x 3 cm dimensions so that the laser energy entered and exited through one pair of windows separated by 10 cm. The observations of emission were made at a 90 degree angle through a third window. This and the fourth window were also used for absorption experiments, keeping all other experimental conditions the same as in the emission determinations.

Although the mechanism for emission is not yet fully understood, it is obvious that the efficiency of collisional transfer is sufficient to populate a total vibration-rotation band when only a few of its members are completely saturated. Further investigation of fluorescence is planned utilizing longer excitation paths, time resolved relaxation measurements, and higher resolution infrared instrumentation.

A. M. Ronn prepares a carbon dioxide laser for fluorescence experiments.



[1] See **Laser induced infrared fluorescence**, by A. M. Ronn, J. Chem. Phys. Letter (to be published).



Asymmetrical curves for the heat capacity of a titanium-nickel alloy indicate a higher than first order transition as was previously suspected. The transition temperature shifted to a lower temperature each time the specimen was heated.

ENGINEERING DATA OBTAINED FOR TITANIUM-NICKEL ALLOY

Heat Capacity Determined From 25 to 220 °C

■ Titanium-nickel alloys have favorable properties for many applications. Their resistance to stress-corrosion cracking makes them ideally suited to marine environments. Their high-hardness and nonmagnetic properties make them suitable for bearings, handtools, cutlery, and instrument components. The 50-50 TiNi alloy is also of special interest because of the solid-state transition it undergoes when heated above room temperature. A wire made from this alloy, for example, has the odd property that when bent at room temperature it will straighten as it is heated through the transition range. Understanding this transition is one of the fundamental problems associated with TiNi alloys.

Because of this alloy's potential for engineering applications as well as the fundamental interest in the nature of the transition, H. A. Berman and E. D. West of the NBS Institute for Basic Standards in cooperation with A. G. Rozner of the Naval Ordnance Laboratory investigated the specific heat of a TiNi alloy (50 atomic percent of each element) in the temperature range from 25 to 220 °C.[1] The entropy of transition, which is important for understanding the nature of the transition, was found from the heat capacity data to be 2.76 entropy units (11.5 J/mol-K).

Heat capacity measurements in the transition region are especially difficult for this type of alloy because of the slow approach to thermal equilibrium after the alloy is heated. However, the NBS adiabatic calorimeter is especially applicable for studying the transition region because slow changes in the specimen can be followed by observing the calorimeter temperature, which changes slowly in a predictable way unless influenced by an energy change in the specimen itself.

The NBS study, therefore, consisted of determining the amount of energy required to raise the calorimeter and its contents by a small amount, usually about 10 deg C, but as little as 0.8 deg C in the transition region. The experiments were then repeated with the calorimeter empty. The heat capacity of the sample was obtained from the difference between smoothed heat capacity curves for the calorimeter with the sample and for the empty calorimeter. The curves obtained by this method were asymmetrical, thus showing unambiguously that the transition is higher than first order.

For study, a cast TiNi billet was prepared at the Naval Ordnance Laboratory by nonconsumable arc melting. The billet was then hot-swaged, annealed for 2 hours at 800 °C, and cooled overnight at a vacuum of 10^{-6} torr. A cylindrical specimen approximately 1.0 cm in diameter and 4.5 cm long was then ground from the billet.

Three series of measurements were made on the sample. The first extended from 128 to 218 °C. The calorimeter was then allowed to cool to room temperature for the second series from 25 to 154 °C. Following this, the calorimeter was again cooled to room temperature and a single experiment was made from 34.9 to 151.0 °C to obtain the overall heat of transition. The calorimeter was again cooled for the third series, which extended from 40 to 179 °C.

Analytical representations for heat capacity as a function of temperature were obtained by fitting theoretical equations to the data points using the method of least squares. The standard deviation of the residuals, that is, the differences between individual measurements and the fitted curves, was within 0.13 percent for the empty calorimeter in the temperature range from 30 to 145 °C and within 0.14 percent for the 145 to 220 °C range. For the full calorimeter the standard deviation figures were within 0.14 percent for the range below the transition region and within 0.16 percent at temperatures above this region.

Values for the heat of transition were obtained by two methods: from the energy increments of the long-

continued

ALLOY *continued*

range experiment and by calculating the area under the curves of observed heat capacities during the transition for the second and third series. The three values for the heat of transition were: second series, 4150 J/mol; single experiment, 3831 J/mol; and, third series, 3375 J/mol.

[1] **Anomalous heat capacity of TiNi**, by H. A. Berman, E. D. West, and A. G. Rozner, J. Appl. Phys. **38**, 4473 (1967).

ION CHAMBER PROVIDES CALIBRATED ULTRAVIOLET DETECTION

L. R. Canfield examines a double ion chamber prior to its installation in experimental apparatus to evaluate its use as an ultraviolet detector.

■ Ultraviolet detectors of spectral irradiance—and means for their calibration—have recently become of increased importance, particularly in space applications. The detector most commonly used for spectral measurements in the extreme ultraviolet is a phosphor-sensitized photomultiplier which is calibrated using a thermopile as a reference. However, disadvantages have long been known to be associated with the comparison calibration in that a preliminary study is required of the variation in sensitivity over the active area of the thermopile, and of the variation in the intensity through a cross section of the incident beam. The thermopile must also be corrected for the loss of sensitivity due to energy being carried away by photoejected electrons. In addition, thermopiles lack sufficient sensitivity to enable their use on more than a very few of the spectral lines that are typically available in the laboratory.

In a recent study[1] aimed at improving radiometric accuracy in the extreme ultraviolet, L. R. Canfield, R. G. Johnston, K. Codling, and R. P. Madden of the NBS Institute for Basic Standards investigated a detector based on the photoionization of rare gases.

The use of such an ion chamber for absolute radiometry was proposed in 1964.[2] It offers relatively simple operation and sensitivity greatly improved over that of a calibrated thermopile. The detector measures the ion current created by the photoionization of a rare gas by extreme ultraviolet photons. The ion current is a direct measure of the photon flux. The only requirement needed to assume inherent calibration of such an ion chamber is that each absorbed photon causes the formation of one singly charged ion. The observed ion current may then be equated to a specific value of spectral irradiance, independent of pressure and absorption cross section. In the NBS study a comparison was made between a thermopile and an ion chamber using extreme ultraviolet irradiance of 584 Å and 735 Å wavelengths from a vacuum monochromator. The thermopile, which had previously been calibrated against an NBS standard of total irradiance, was interchanged with the ion chamber in a series of measurements at each wavelength. The corrections previously discussed were applied to the thermopile data.

Argon served as the gas in the double-ion chamber. Several argon pressures as well as differing light source currents were used for each set of measurements.

The results indicated that the ion chamber and the calibrated thermopile are in agreement within the limits of estimated experimental error (within 3 percent).

This successful comparison greatly increases the confidence which can be placed in the rare-gas double-ion chamber for the determination of absolute flux levels in the extreme ultraviolet. A significant advantage resulting from this increased confidence is that any laboratory desiring to determine absolute values of spectral irradiance in the spectral region of noble gas photoionization may construct such a device and perform the determination without reference to another detector or source of calibration.

[1] See, **Comparison of an ionization chamber and a thermopile as absolute detectors in the extreme ultraviolet**, by L. R. Canfield, R. G. Johnston, K. Codling, and R. P. Madden, Appl. Opt. **6**, 1886 (Nov. 1967).

[2] J. A. R. Samson, J. Opt. Soc. Am. **54**, 6 (1964).

NBS TO AID NATIONAL CONFERENCE OF STATE BUILDING ADMINISTRATORS

■ The NBS Institute for Applied Technology has agreed to provide, primarily through its Building Research Division, both technical and secretarial assistance to a proposed new organization tentatively called the National Conference of States on Building Codes and Standards. NBS will serve the Conference in a capacity similar to that now offered the widely respected National Conference on Weights and Measures,[1] which was founded 62 years ago.

The establishment of a National Conference of States on Building Codes and Standards was approved at a meeting held on November 20, 1967, in Madison, Wis., at the invitation of Wisconsin Governor Warren P. Knowles. Attending were representatives of 19 States, the building industry, the Advisory Commission on Intergovernmental Relations, and the National Bureau of Standards. At the next meeting, scheduled for May 1968, delegates hope to agree on objectives, organizational structure, and procedures of the new Conference.

The interest of the Bureau in this new organization stems from its well-known support of voluntary national standards, particularly in the building field. The Bureau's Building Research Division participates through membership of its staff on the committees of many standards-producing organizations that include the American Concrete Institute; National Fire Protection Association; American Society of Heating, Refrigerating, and Air-Conditioning Engineers; American Society for Testing and Materials; United States of America Standards Institute (formerly American Standards Association); and others, both domestic and foreign.

Much of the building research work at the Bureau is directed toward development of test methods, particularly those of the performance type, which are necessary to assess the specified properties of building materials, components, and systems. One of the most important uses of building standards and test evaluation procedures is in the improvement of building codes. The Bureau therefore extends its interest in standards to the code field and to the voluntary standardizing process itself, including the relationships among standards that are used in the final code documents.

Much has been written, particularly in recent years, about the building code system in the United States wherein most county or city code jurisdictions adopt provisions of one of the four model codes, usually with modifications to suit local customs and political pressures. The present system often fails to present a large enough market area with uniform requirements to justify the research and promotion efforts required to develop better or less expensive building systems. By tradition, codes have been of the prescription rather than of the performance type, and this too has led to difficulty in the acceptance of building innovations. The Bureau, therefore, is interested in the improvement of national building standards and performance evaluating procedures useful to those responsible for the promulgation, implementation, and enforcement of codes.

A number of groups are studying and have studied the building code problem. One very important study was made by the Advisory Commission on Intergovernmental Relations, which deals with problems common to different levels of government. One of the recommendations of the ACIR is that the Federal Government take steps to bring about uniformity in its own construction efforts. Several ACIR code improvement recommendations dealt with actions to be taken by the States. NBS felt that it might assist the States by providing a forum for discussion of mutual problems and solutions, patterned after the National Conference on Weights and Measures, which has served the States and other jurisdictions so very effectively over the years.

On May 25 and 26, 1967, a meeting was held at the Bureau with representatives from seven States. Included were such people as a State fire marshal, a State architect, and a State Commissioner. This somewhat diverse group concurred with NBS on the potential usefulness of a National Conference of State Building Officials. It was agreed that the need and climate existed now for joint action involving the solution of building code problems within and among the States, and that the solution to such problems would, as a byproduct, assist in the development of uniform construction programs. All of those present were enthusiastic in regard to the potential national conference, and therefore, at the meeting held in Madison, it was decided to proceed with the formal organization of the Conference.

The Conference will be an organization of representatives of State governments who will control its scope and operations. The Bureau will furnish the secretariat and will provide, as its resources permit, a research effort directed toward solution of code problems brought to it through the Conference. The role of NBS in this organization will be similar to its role in the National Conference on Weights and Measures. Where possible, the Bureau will also assist the committees established by the Conference in making recommendations on a range of subjects common to various State organizations.

continued

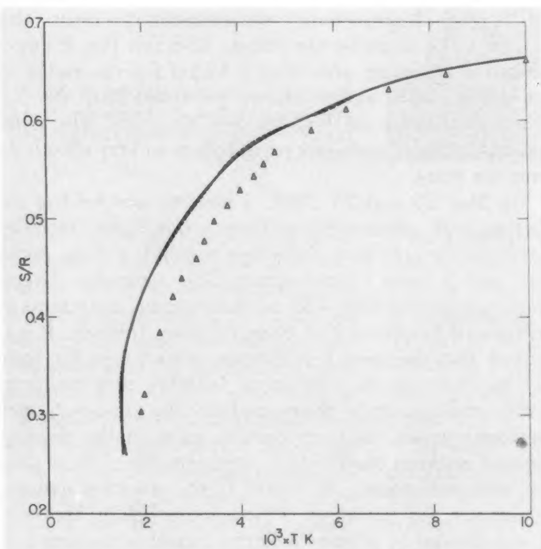
STATE BUILDING *continued*

The principal contact between the Bureau and the Conference will be the Codes and Standards Section within the NBS Building Research Division. This section is now headed by Gene A. Rowland, the former Commissioner of Wisconsin's Department of Industry, Labor, and Human Relations. Mr. Rowland brings to the Bureau a wide range of experience in the building construction industry and its related problems, with particular emphasis on building codes. While Commissioner, Mr. Rowland was elected temporary chairman of the Conference at its November meeting in Madison.

The Conference has a great potential for promoting national building standards, through the performance concept, and a partnership with the Bureau in developing evaluating techniques. The realization of its full potential presents an important challenge to the State leaders involved and to the Bureau's Building Research Division.

[1] NBS also sponsors and provides the secretariat for the National Conference of Standards Laboratories, an organization of industrial, academic, and government laboratories concerned with standards of physical measurement.

CRYOGENIC TEMPERATURE MEDIUM INVESTIGATED



Absolute temperature versus entropy of cerous magnesium nitrate at cryogenic temperatures. The full line is a smoothed curve of raw data (dashed line); the triangles are recent data of other workers.[3]

■ In the last twenty years, studies at extremely low temperatures have become a major area of research. Experimental investigations of superconducting metals, superfluid helium, radioactivity, nuclear magnetism, semiconductors, and many others are being made in this temperature region to determine the validity of important physical theories. In addition, studies of the low-temperature behavior of materials are becoming increasingly important in industry and technology, and particularly in the space program.

A particular inorganic salt, cerous magnesium nitrate, CMN, has been gaining widespread use as a medium for obtaining and determining very low temperatures (below 2 K). As its paramagnetic susceptibility follows the Curie law* down to approximately 0.006 K, CMN appears well suited for thermometric indication based on magnetic susceptibility. Previous studies on this salt, however, have provided a temperature scale that has been of somewhat questionable reliability. This has led R. P. Hudson and R. S. Kaeser of the NBS Institute for Basic Standards to re-investigate the CMN temperature scale.[1] Results obtained in the study disagree with earlier reported results [2] but are in reasonable agreement with recent work by a different method.[3]

In the NBS study, low temperatures were obtained by adiabatic demagnetization. Temperatures through the Curie law range were determined from susceptibility measurements made by means of a magnetic thermometer in which the CMN specimen formed the core of a mutual inductance. Inductance was measured by means of a Hartshorn bridge. In the magnetic thermometer, inductance between the primary and the secondary coils is a linear function of magnetic susceptibility. The CMN specimen was mounted at the top of a low-thermal conductivity support within a glass vacuum container. The support was anchored to the center of a ground-glass plug which sealed off the vacuum container. This assembly was contained in a liquid helium Dewar,

which in turn was immersed in a liquid nitrogen Dewar.

In the temperature region below that at which the Curie law holds, temperature measurements are made by the same experimental procedure but are referred to as magnetic temperatures (T^*). These temperatures must be converted to absolute temperatures by demagnetizations, heating, and calculations using known thermodynamic relationships. In this procedure, the entropy at the start of each demagnetization is determined from known conditions of temperature and field. The demagnetization is then performed with no change of entropy and the susceptibility measured. This yields a relationship between the salt entropy and magnetic susceptibility. Next, heat is added to the salt and the temperature rise is measured. This yields a relationship between Q, the heat added, and T^*, the magnetic temperature. From these two relationships the absolute temperature may be calculated.

For heating the specimens after cooling, a gamma-ray source was used consisting of 250 mCi cobalt-60 in the form of 16 cobalt wires placed coaxially about the specimen in a cylindrical brass container. The CMN specimens were heated at a rate of approximately 0.0002 joule per minute, which is about 50 times greater than the natural heat leak.

Large single crystals of CMN, ground to the desired shapes (spherical and ellipsoidal), were used in the study. The constituent cerous and magnesium nitrates were the purest commercially available and the CMN was further recrystallized several times before final growth was initiated. A quantitative analysis of the CMN was obtained for identification of those impurities present at the parts per million level.

The study has confirmed the range of the Curie law (down to 0.006 K). The region of rapid fall-off of entropy was found to be at 0.0015 K rather than the 0.003 K originally reported.[2] The salt exhibited practically constant susceptibility, and hence constant magnetic temperature, for all entropies smaller than 0.38 R; thus, in the latter range, magnetic susceptibility is no longer a useful thermometric indicator.

Specific heat varied as a function of $(T^*)^{-2}$ for magnetic temperatures greater than 0.006 K. There was found, however, anomalous behavior above 0.03 K. Here, there was a departure from the linear dependence of mutual inductance on time for continuous gamma irradiation, indicating the appearance of an additional heat capacity. This anomaly is unexplained, but is apparently nonmagnetic in origin.

A particularly intriguing feature of the properties of CMN has appeared in research employing as a magnetic thermometer cylindrical specimens of the salt made from compressed powder.[4] Strong, if indirect, indications were obtained that such specimens exhibit Curie law behavior right down to 0.002 K. Perhaps it will be possible to explain this behavior in terms of the particular shape of the specimens and the extreme magnetic anisotropy of CMN, but further research will be required to elucidate this.

[*] The Curie law may be stated as $\chi = \frac{C}{T}$, where χ is the magnetic susceptibility, T is the absolute temperature, and C is the Curie constant.

[1] For further details, see **Cerous magnesium nitrate: A magnetic temperature scale 0.002–2 °K,** by R. P. Hudson and R. S. Kaeser, Physics **3**, No. 2, 95–113 (1967).

[2] J. M. Daniels and F. N. H. Robinson, Phil. Mag. **44**, 630 (1953).

[3] R. B. Frankel, D. A. Shirley, and N. J. Stone, Phys. Rev. **140**, A1020 (1965); **143**, 334 (1966).

[4] W. R. Abel, A. C. Anderson, W. C. Black, and J. C. Wheatley, Physics **1, No. 6, 337–387** (1965).

RESEARCH ASSOCIATE COMPLETES ASSIGNMENT AT NBS

Allen V. Astin, NBS Director, presents a Certificate of Appreciation to Carl F. Shelton, as (from left to right) Alvin McNish, Frank Studer, and Louis Barbrow, look on. Mr. Shelton, under sponsorship of IBM Corp., has just completed a one-year Research Associate Program at the Bureau. The citation on the certificate which he received on January 4, 1968, reads: ". . . for his contribution to knowledge of the spectral emission characteristics of NBS Standard Reference Phosphors, and to a better understanding of the effects of ultraviolet excitation on these phosphors."

Apparatus to detect particle contamination in liquids with a laser beam. The photomultiplier detector and parabolic mirror are at the left (inside box). On the right (outside box) is the laser source. The bright, vertical line between the mirror and detector is the laser beam scattered by suspended particles in the test solution.

LASER used to detect small particles in liquids

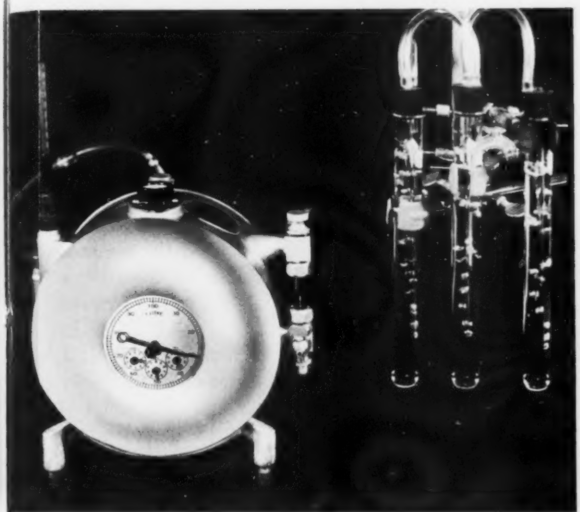
■ A laser light scattering apparatus constructed at the National Bureau of Standards provides a rapid and convenient, yet highly sensitive, method for determining extremely small concentrations of solid particles suspended in liquids. The method should be of broad utility in such fields as water and air pollution, medical and bacteriological research, particle-free lubrication, and manufacturing process control in microminiaturization. The work was described in a paper,[1] by D. H. Freeman and E. C. Kuehner of the NBS Institute for Materials Research, presented on February 2, 1968, before the New York Academy of Sciences at a Conference on Liquid-Borne Particle Metrology.

While the laser scattering technique is applicable to a number of fields, it was devised primarily to solve problems that have arisen in analytical chemistry. In the past several years, analytical chemical measurements have become sensitive to the effects of extremely small quantities of chemical contamination. One of the more difficult problems has been the presence in liquids of solid particles that are not large enough to be removed by conventional filters and whose concentration is below the level of detection by the usual methods of measurement. Typically the size of the suspended particle is less than a thousandth of a centimeter. Elaborate and cumbersome apparatus and techniques have been required to detect such particles in the small concentrations that are often troublesome. The NBS scientists therefore set out to build an apparatus for this purpose that would not require much time for operation and maintenance.

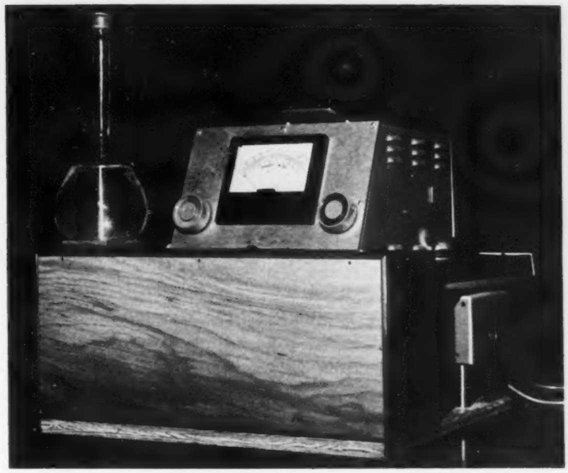
The use of a laser for particle contamination measurement is based upon the fact that such small particles are a billion times more effective in scattering light than are the liquids in which they are suspended. The laser has the important advantage of providing a very highly concentrated beam of light, making feasible the use of small sample volumes and a relatively simple detection device such as a photomultiplier.

The apparatus uses an optical train to pass a beam from a continuous-wave helium-neon laser through the central axis of a glass tube holding the liquid sample. A photomultiplier tube placed to one side of the sample tube measures light scattered perpendicularly away from the beam path. A concave mirror on the opposite side of the sample tube is used to help return back-scattered light toward the photomultiplier detector.

To calibrate the apparatus a series of standard solutions was prepared in which the weight concentration of



Metered amounts of air-borne dust are trapped by passing the air into pure water, which is then measured using the laser light scattering technique.



Before a solution suspected of being contaminated is actually measured for particle contamination, a quick visual test may be made by passing a laser beam through the solution. If light scattering is evident, as in this case, then the NBS technique for measuring the amount of contamination is employed. This sample of a water-latex suspension was found to contain 10 ppm of polystyrene latex particles.

suspended particles was known. From the measured light scattering of the solutions of known concentration, it was possible to estimate the corresponding particle concentration in various samples.

The method was found to be so sensitive that concentrations corresponding to the ultimate limit of light scattering were readily obtained. For practical purposes this concentration is a few hundred particles per milliliter, or one part by weight of particles to a billion parts by weight of the liquid.

The results of a survey of various liquids demonstrated that the method is broadly suited to the study of particulate contamination. For example, it readily distinguishes between the particle concentrations by weight in distilled water (0.000 000 1 percent), in drinking water (0.000 002 0 percent), and in river water or tidal estuary water (0.000 01 percent). Microfilters are available for removing trace quantities of particles, although no simple test of their efficacy has been made. In the present study it was found that such filters tend to be contaminated and are not effective until this contamination is removed.

After a routine procedure had been developed for these measurements it was possible to show that the laser technique has application to problems in a wide variety of areas. For example, although it provides no direct way of determining particulate contamination in solid materials, the solids may be dissolved in suitable liquids and the concentration of undissolved particles then measured. In a number of instances the contamination of such chemicals was found large enough for serious concern. The simple error of leaving an open vessel exposed to the laboratory air was found to cause a significant increase in particle contamination.

The observed fallout of particles from the laboratory air suggested the use of the method to measure the concentration of atmospheric dust. Measured volumes of air were slowly bubbled through a liquid so that all the atmospheric dust was trapped. This made possible a measurement of the amount of dust per unit mass of air, and that sample of the air was found to be five times as contaminated with dust as ordinary tap water. This measurement suggests the desirability of directly measuring the residual air pollution in atmospheres that have already been carefully screened to remove the suspended particle contamination.

[1] Laser detection of small particules in liquids, by D. H. Freeman and E. C. Kuehner, presented before the Conference on Liquid-Borne Particle Metrology, New York, N.Y., Feb. 1–2, 1968. To be published in **Liquid-Borne Particle Metrology**, Ann. N.Y. Acad. Sci.

STANDARDS AND CALIBRATION

CALIBRATION SERVICE FOR NONREFLECTING WAVEGUIDE PORTS

The Radio Standards Laboratory (Boulder, Colo.) of the NBS Institute for Basic Standards announces a calibration service for the evaluation of nonreflecting waveguide ports, including "matched loads." The frequency coverage of the service is continuous from 2.6 GHz to 18.0 GHz and includes six waveguide sizes: WR284, WR187, WR137, WR112, WR90, and WR62.

The term *nonreflecting* as used here indicates that the waveguide port has been designed or adjusted with the intent to produce a reflection coefficient magnitude, $|\Gamma|$, equal to zero. Although most waveguide ports for such applications cannot produce a reflection coefficient magnitude identically equal to zero, their reflection coefficient magnitudes often approach zero very closely. Useful information is gained by evaluating the extent to which this is accomplished. There are many applications in the laboratory for nonreflecting waveguide ports, including the termination commonly referred to as a "matched load."

Considerable care must be exercised in the use of nonreflecting waveguide ports by keeping the mating connector flange surfaces smooth and clean beyond that of usual practice. Accurate alinement of the interior surfaces of the joining waveguides at the flange junction also is very important. The back of the flange which makes contact with the connecting bolts should be nominally flat and free of soft materials, including paint. These precautions must be observed when using a nonreflecting waveguide port in a precision measurement system.

Nonreflecting waveguide ports can be measured with an uncertainty of $\pm(0.000\ 13+0.0032\ |\Gamma|)$, where $|\Gamma|$ is the numerical value of the measured magnitude. The present reflection calibration system can be used to measure reflection coefficient magnitude as small as 0.0001 with the above uncertainty.

Reflections may be introduced in a measuring system by misalinement of a nonreflecting waveguide port or by foreign matter and mechanical imperfections at the flange junction, and the uncertainty of calibration can be exceeded if precautions are not taken.

STANDARDS LABORATORIES TO MEET IN AUGUST

The 1968 Standards Laboratory Conference will be held August 26–29 at the Boulder Laboratories of the National Bureau of Standards. As in the past, the conference is being sponsored and presented by the National Conference of Standards Laboratories (NCSL).

NCSL is a nonprofit laboratory-oriented organization to promote cooperative efforts toward solving the common problems faced by standards laboratories in their organization and operation. It was established in 1961 under sponsorship of the National Bureau of Standards. Its membership consists of academic, scientific, industrial, commercial, or governmental laboratories concerned with the measurement of physical quantities, the calibration of standards and instruments, and the development of standards of laboratory practice. It also provides liaison with technical societies, trade associations, and educational institutions interested in these activities.

General theme of the conference this year will be: Making Valuable Measurements. E. J. Arsenault of the General Electric Company (Philadelphia, Pa.) is Conference Coordinator; and the technical program is being organized by J. L. Hayes, Metrology Engineering Center, Naval Plant Representative, 1675 West Fifth Ave., P.O. Box 2507, Pomona, Calif. 91766. General questions about conference arrangements should be directed to George Goulette, University of Colorado, 328 University Memorial Center, Boulder, Colo. 80302.

IMPROVED CALIBRATION ACCURACY FOR COAXIAL IMPEDANCE (1–8 GHz)

Accuracies of impedance measurements in coaxial-line systems have been improved significantly in recent years [1] by the Radio Standards Laboratory (Boulder, Colo.) of the NBS Institute for Basic Standards, and others. This improvement came primarily from the development of precision coaxial-line standards and precision coaxial connectors.[2] These developments have in turn contributed toward improving measurement capabilities of coaxial slotted-line systems to the extent that very accurate measurements are now possible. Errors originally introduced by structural defects of slotted lines have been minimized by the use of precision made, coaxial slotted lines. Refinements in measurement technique have helped, in part, to reduce some systematic errors.

These improvements, along with other good practices, make the measurement possible of VSWR (Voltage Standing Wave Ratio) up to 8 GHz with an uncertainty in the range of 0.1 to 1 percent. The phase of the reflection-coefficient magnitude can be measured with an uncertainty ranging from 0.1 degree to approximately 1 degree. These uncertainties apply for coaxial impedance standards equipped with the 14-mm precision coaxial connector,

where $1 \leq$ VSWR ≤ 2, referred to 50 ohms. Coaxial impedance standards equipped with other than precision connectors and having higher VSWR's can also be measured, but with reduced accuracy.

Error equations, derived by W. E. Little,[3] were used to determine the uncertainty in measurement of VSWR and phase angle as a function of the errors due to:

(a) Residual VSWR
(b) Probe instability
(c) Detector insensitivity
(d) Inability to determine the voltage minimum of the standing-wave pattern
(e) Generator frequency and power instabilities.

To verify the measurements made on the slotted-line system, an intercomparison was made of VSWR's (1.04 to 2.0) of transfer standards measured by slotted-line techniques and by a tuned reflectometer. Maximum differences between measurements on the slotted line and the tuned reflectometer were less than 0.2 percent at 4 GHz and less than 0.4 percent at 6 GHz.

CALIBRATION SERVICE FOR MICROWAVE POWER IN WR284 WAVEGUIDE (2.60–3.95 GHz)

The Radio Standards Laboratory (Boulder, Colo.) of the NBS Institute for Basic Standards announces a calibration service for the measurement of effective efficiency and calibration factor of bolometer units and bolometer-coupler units in WR284 waveguide. Although calibrations can be performed at any frequency in the recommended range of the waveguide (2.60–3.95 GHz), it is more economical to the customer if calibrations are performed at the suggested[4] frequencies of 2.85, 3.25, and 3.55 GHz.

Interlaboratory standards consisting of bolometer units and bolometer-coupler units calibrated at NBS form the principal link to the NBS Reference Standards of microwave power. These calibrated bolometric units are then used to calibrate other power measuring devices in various standards laboratories; they can also serve as a check upon the operation of power measuring systems.

The quantities measured in these services have the following definitions:[5]

Effective efficiency of bolometer units: The ratio of the substituted d-c power in the bolometer unit to the microwave power dissipated within the bolometer unit.

Calibration factor of bolometer units: The ratio of the substituted d-c power in the bolometer unit to the microwave power incident upon the bolometer unit.

Calibration factor of bolometer-coupler units: The ratio of the substituted d-c power in the bolometer unit on the side arm of the directional coupler to the microwave power incident upon a nonreflecting load attached to the output port of the main arm.

Because of certain limitations in adapting the microcalorimeter to the measurement of power in larger waveguide sizes, the effective efficiency and calibration factor of bolometer units in WR284 waveguide are measured somewhat indirectly, using a working standard that has been calibrated by the impedance method.[6, 7, 8] The impedance technique yields a direct measurement of the *efficiency* of a bolometer unit, which is defined as the ratio of the microwave power absorbed by the bolometer element to the microwave power dissipated within the bolometer unit. However, this efficiency measurement usually is of special interest only. The efficiency of the specially designed working standard, which has been measured by the impedance method, can be converted into effective efficiency by taking into account the dc-rf substitution error.[9, 10] The effective efficiency determined for the working standards then can be used as a basis for measuring the effective efficiency and calibration factor of other bolometer units and bolometer-coupler units having either barretter or thermistor elements.

The effective efficiency of a bolometer unit in WR284 waveguide can be measured with limits of uncertainty equal to ± 1.2 percent. In determining the calibration factor of a bolometer unit, the reflection coefficient of the bolometer unit being calibrated must be taken into account. With a reflection coefficient of 0.2, limits of uncertainty for the calibration factor are ± 1.6 percent. If the reflection coefficient is nearly zero, the limits of uncertainty for the calibration factor of a bolometer unit are again ± 1.2 percent. For each of these measurements, the bolometer element can be of either the barretter or thermistor type having a normal operating resistance of either 100 or 200 ohms at a bias current in the range 3.5 to 15 mA. Bolometer units of either the fixed-tuned or untuned broadband type are acceptable for calibration. Power measurements on bolometer units normally are made at a power level of 10 milliwatts.

The calibration factor of bolometer-coupler units can be measured in WR284 waveguide with limits of uncertainty equal to ± 1.1 percent. Bolometer units of either the fixed-tuned or untuned broadband type must be permanently attached to the side arm of the directional coupler. The coupling ratio can range from 3 to 20 dB. The coupler should have a directivity of no less than 40 dB and a VSWR no greater than 1.05 at the input and ouput ports of the main arm of the coupler. The calibration factor of bolometer-coupler units normally is measured at a power level of 10 milliwatts for a coupling ratio of 3 dB, 1 milliwatt for a coupling ratio of 10 dB, and 0.1 milliwatt for a coupling ratio of 20 dB.

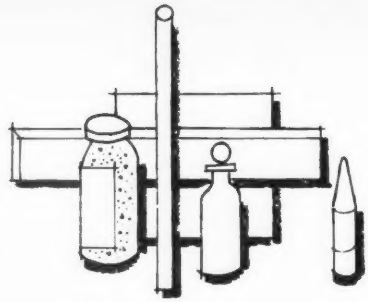
STANDARD FREQUENCY AND TIME BROADCASTS

WWV—2.5, 5.0, 10.0, 15.0, 20.0, and 25.0 MHz
WWVH—2.5, 5.0, 10.0, and 15.0 MHz
WWVB—60 kHz

Radio stations WWV (Fort Collins, Colo.) and WWVH (Maui, Hawaii) broadcast signals that are kept in close

continued on p. 89

STANDARD REFERENCE MATERIALS



Standard Reference Materials are well-characterized materials disseminated by NBS to be used in calibrating and evaluating measuring instruments, methods, and systems or to produce scientific data that can be referred readily to a common base. These materials are certified for chemical composition or for a particular physical or chemical property. They are used on-site in science and industry for calibrating the instruments and methods used for production and quality control of raw materials, chemicals, metals, ceramics, fuels, and radioactive nuclides in manufacturing processes and in research. This column regularly reports on the issuance of new and renewal Standard Reference Materials and on latest developments in the Standard Reference Materials Program.[1]

CHROMIUM STEEL STANDARD

A new chromium steel standard, containing 1 percent nominally of carbon, manganese, and chromium, has recently become available. Designated NBS Standard No. 163, it is sized between 25 and 60 mesh sieves and issued in 100 g units. This standard was prepared primarily for use in the steel industry for standardizing chemical methods of analysis. It is useful for both production control and customer acceptance tests. The price is $35 per unit.[2]

NBS Standard No. 163 was prepared by a pre-alloyed powder metallurgical process which included argon atomization and hydrogen annealing. It is the first ferrous-base standard to be prepared by this process.

An extensive investigation has been made of its homogeneity, and particle size and shape distribution. The homogeneity in the final product was found to be entirely acceptable. In general, the change from the usual chip samples to the spherical shape does not seem to pose problems for most analysts. In fact, after working with the powdered material, some laboratories prefer it to the chip form.

Because of the nature of this new standard, a comprehensive program of certification was performed at NBS and at 12 cooperating laboratories. The standard is issued with a final Certificate of Analysis covering its content of carbon, manganese, phosphorus, sulfur, silicon, copper, nickel, chromium, molybdenum, and nitrogen.

The powder metallurgical approach for the preparation of certain metal standard reference materials offers important advantages over the usual mode of preparation. For example, the process is capable of producing a highly homogeneous material. In addition, significant production savings should be forthcoming, once the process becomes routine. With conventional methods of preparing standards in chip form, the starting material is melted in a furnace, cast into an ingot, fabricated into round billets, and then chipped with machine tools. The pre-alloyed powder approach used for Standard No. 163 eliminates these intermediate steps with consequent substantial savings. For materials that are especially difficult to machine into chip form, such as some grades of stainless steel, this approach should provide even greater savings, in both time and money.

ZINC OXIDE

Zinc oxide is used on a worldwide basis by the rubber and carbon black industries as an activator in the production of rubber products.

Rubber recipes are evaluated by preparing a vulcanizate and subjecting it to certain tests. For these evaluations, not only the rubber and sulfur, but also the activator, the accelerator, and the filler must react in a known way to processing conditions.

The NBS Standard Reference Materials for the rubber industry include both rubbers and compounding ingredients of which zinc oxide, NBS Standard No. 370, is one. The stock of this compound had been exhausted. The renewal lot, NBS No. 370d, is now available and may be purchased in units of four 2000-gram cans.[2]

This material is of exceptional purity, produced to specifications that yield a product of uniform surface area from particle to particle. The chemical and physical properties of No. 370d resemble very closely those of its predecessor, No. 370c, and its action as an activator is very similar to that of No. 370c.

CARBON-14 LABELED SUGARS AND RARE SUGARS

The prices of a number of the carbon-14 labeled sugars available from the NBS Office of Standard Reference Materials have recently been reduced. These carbohydrate research materials were synthesized in NBS laboratories by H. S. Isbell and his associates. The accompanying table lists the name of each compound available, the number of microcuries of activity per package, the NBS standard number, and the price.[2]

CARBON-14 LABELED SUGARS [2]

Terminal Carbon-14 Sugars

NBS No.	Kind		Price
1526	D-Arabinose-1-	;200 μCi	$150
		; 60 μCi	58
1527	L-Arabinose-1-	;200 μCi	150
		; 60 μCi	58
1528	D-Galactose-1-	;300 μCi	150
		;100 μCi	62
1529	D-Galactitol-1-	;200 μCi	150
		; 60 μCi	58
1530	D-Glucose-1-	;300 μCi	150
		;100 μCi	62
1531	D-Glucitol-1-	;200 μCi	150
		; 60 μCi	58
1532	Lactose-1-	;120 μCi	150
		; 40 μCi	62
1533	D-Lyxose-1-	;200 μCi	150
		; 60 μCi	58
1534	Maltose-1-	;120 μCi	150
		; 40 μCi	62
1535	D-Mannose-1-	;300 μCi	150
		;100 μCi	62
1536	D-Mannonic-1-(Lactone)	;100 μCi	62
1537	D-Mannitol-1-	;200 μCi	150
		; 60 μCi	58
1538	L-Rhamnose-1-	;120 μCi	150
		; 40 μCi	62
1539	D-Ribose-1-	; 60 μCi	58
1540	D-Xylose-1-	;150 μCi	150
		; 50 μCi	62

Interior Carbon-14 Sugars

NBS No.	Kind		Price
1551	D-Arabinose-5-	;120 μCi	$150
		; 40 μCi	62
1552	D-Galactose-2-	; 80 μCi	140
		; 25 μCi	56
1553	D-Glucose-2-	; 80 μCi	140
		; 25 μCi	56
1554	D-Glucose-6-	;200 μCi	150
		; 60 μCi	58
1555	D-Glucurone-6-	;200 μCi	150
		; 60 μCi	58
1556	D-Glucurone-6-(Na salt)	;200 μCi	150
		; 60 μCi	58
1557	D-Xylose-2-	; 80 μCi	140
		; 25 μCi	56
1558	Dextran; NRC-1	;150 μCi	60
1559	Dextran; NRC-2B	;150 μCi	60
1560	Dextran; NRC-3	;150 μCi	60
1561	Dextran; NRC-4	;150 μCi	60
1562	Inulin	;500 μCi	60

The specific radioactivity of each compound offered is supplied the user; each is in the range of 1–9 μCi per mg, except for the dextrans for which the activity ranges to as low as 0.2 μCi per mg because of their high molecular weights. The labeled compounds are purified and rechecked for radiochemical purity before shipment.

Four nonradioactive carbohydrates are also being made available as standard reference materials. They are rare compounds that are not available from commercial sources.

NBS Standard No. 1591, 1,2-*O*-Isopropylidene-β-L-Idofuranose, from which the rare, somewhat unstable hexose, L-idose, can be easily obtained, is available in quantities of 15 mg units. This standard is priced at $30 per unit. NBS Standard No. 1592, 2,3-*O*-Isopropylidene-β-D-*threo*-Pentulose ("monoacetone-D-xylulose") is a stable crystalline derivative, which can be readily converted into "D-xylulose," and is available in a package containing 50 mg for $30. NBS Standard No. 1593, *levo*-Inositol, and NBS No. 1594, Quebrachitol (a monomethyl ether of *levo*-inositol), are also available. These standards are sold for $30 each. NBS No. 1593 weighs 0.25 gram and NBS No. 1594 weighs 0.5 gram.

Two other interesting compounds that will shortly be made available are 1,2:3,4-di-*O*-isopropylidene-D-*erythro*-pentulofuranose ("diacetone-D-ribulose") and 1,2:4,5-di-*O*-isopropylidene-D-*ribo*-hexulose ("diacetone-D-psicose").

[1] For a complete list of Standard Reference Materials available from NBS, see **Standard Reference Materials: Catalog and Price List of Standard Materials Issued by the National Bureau of Standards**, NBS Misc. Publ. 260 (1968 edition), for sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for 45 cents. Quarterly insert sheets which update Misc. Publ. 260 are supplied on request.

[2] These standards may be purchased from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C. 20234.

NEWS

This column regularly reports significant developments in the program of the National Standard Reference Data System. The NSRDS was established in 1963 by the President's Office of Science and Technology to make critically evaluated data in the physical sciences available to science and technology on a national basis. The System is administered and coordinated by the National Bureau of Standards through the NBS Office of Standard Reference Data, located in the Administration Building at the NBS Gaithersburg Laboratories.

The Document Image Code System

Part of the program of the NBS Office of Standard Reference Data is the development of a General Purpose Scientific Document Image Code (GPSDIC) system. The purpose of a GPSDIC system is to relieve the scientist of the chore of changing his notations to meet arbitrary restraints imposed by machine processing. Such a system has been designed and is currently under development at the Bureau. The design and development of this system is being carried out by Blanton C. Duncan and David Garvin of the Bureau's Physical Chemistry Division in conjunction with the Chemical Thermodynamic Data Group and the Chemical Kinetics Information Center, both of which are at NBS. As designed, the system permits a scientific typescript, with all its complex symbolism and highly structured page format, to be transferred to digital machine form with virtually no limitations on the notations employed.

The system was designed to assure broad applicability by emphasizing: (a) exchange of information via telecommunications devices compatible with the USA Standard Code for Information Interchange (USASCII), (b) design of hardware to permit the use of the proven skills of ordinary scientific typists in the record capture process, (c) exploitation of the capabilities of commercially available extended character high-speed line printers for direct computer output, and (d) publication using this type of machine record as the "typescript" input to computerized typesetting programs.

A significant portion of the system is in daily use utilizing computer programs for the input, editing, and retrieval of records. Such use indicated a need for certain design adjustments, and these have been made. The current effort is directed toward producing the basic detailed descriptive documentation required as part of the specifications in hardware and software procurement.

Because of space limitations, and the fact that parts of the system have been described earlier, a detailed explanation of this system cannot be given here. However, an overall view of the kinds of notational complexity the system is able to handle should be of interest.

The six accompanying design drawings are being used to specify typefaces for a computer printer to be installed shortly at the Bureau. These drawings involve more than the particular specification of a computer printer. They relate to the specification of a class of devices collectively described as General Purpose Scientific Document Writers (GPSDW).

A GPSDW is any output device capable of producing documents at the level of symbolic complexity found in typescripts produced by scientific typists who may overstrike, space by half lines, and use auxiliary typefaces. Besides a typewriter, it may be a line printer, an x-y plotter, a cathode ray display, or any other suitable equipment. An upper boundary of necessary capability lies somewhere between the ability to produce diagrams such as structural formulas from organic chemistry and the ability to produce smooth curves representing mathematical functions. A pragmatic test is whether or not a competent scientific typist could produce the same result.

A typescript page can be viewed as a rectangular array of points. These points are half-spaced vertically (the y-coordinate) and single-spaced horizontally (the x-coordinate). The illustrations that follow show how the concept is applied in detail. Figures 1 and 2 show simple symbols, which are the result of striking a single typeface at an x-y coordinate. A composite symbol is the result of striking two typefaces at the same x-y coordinate. Figure 3 shows part of the set of composite symbols already defined in

continued

0		16	0	32	@	48	P	64	`	80	p
1	!	17	1	33	A	49	Q	65	a	81	q
2	"	18	2	34	B	50	R	66	b	82	r
3	#	19	3	35	C	51	S	67	c	83	s
4	$	20	4	36	D	52	T	68	d	84	t
5	%	21	5	37	E	53	U	69	e	85	u
6	&	22	6	38	F	54	V	70	f	86	v
7	'	23	7	39	G	55	W	71	g	87	w
8	(	24	8	40	H	56	X	72	h	88	x
9	)	25	9	41	I	57	Y	73	i	89	y
10	*	26	:	42	J	58	Z	74	j	90	z
11	+	27	;	43	K	59	[	75	k	91	{
12	,	28	<	44	L	60	\	76	l	92	|
13	-	29	=	45	M	61	]	77	m	93	}
14	.	30	>	46	N	62	^	78	n	94	~
15	/	31	?	47	Ø	63	_	79	o	95	

Figure 1. Simple GPSDIC symbols corresponding to the standard (shift in) USASCII set.

96		112	-	128	∃	144	Π	160	~	176	π
97	¦	113	¦¦	129	∇	145	Θ	161	α	177	θ
98	|	114	•	130	§	146	∫	162	β	178	ρ
99	|	115	•	131	∞	147	Σ	163	•	179	σ
100	_	116	•	132	Δ	148	√	164	δ	180	τ
101	|	117	∿	133	°	149	Υ	165	ε	181	••
102	/	118	←	134	Φ	150	×	166	φ	182	/
103	\	119	→	135	Γ	151	Ω	167	γ	183	ω
104	$\genfrac{}{}{0pt}{}{s}{b}$	120	↑	136	✢	152	◊	168	η	184	χ
105	$\genfrac{}{}{0pt}{}{p}{s}$	121	↓	137	‡	153	Ψ	169	ι	185	ψ
106	∏	122	-	138	Ξ	154	¶	170	ξ	186	ς
107	∑	123	'	139	∝	155	⟨	171	κ	187	∩
108	⊂	124	∧	140	Λ	156	\	172	λ	188	■
109	—	125	⇋	141	∂	157	⟩	173	μ	189	∪
110	⊃	126	∨	142	∈	158	¬	174	ν	190	⌣
111	/	127	⌑	143	●	159	□	175	•	191	

Figure 2. Simple GPSDIC symbols adopted for use as a secondary (shift out) USASCII set.

		ë	2+69	ç	12+67
Ã	33+133	Î	41+62	Ñ	46+94
Â	33+62	Ï	2+41	ñ	78+94
À	33+64	î	62+73	±	11+13
Ä	2+33	ï	2+73	Ø̸	15+47
å	65+133	Ô	47+62	∅	15+79
â	62+65	Ö	2+47	¢	15+67
à	64+65	ô	62+79	≠	15+29
ä	2+65	ö	2+79	∀	54+158
É	7+37	Û	53+62	÷	13+26
Ê	37+62	Ù	53+64	≤	13+28
È	37+64	Ü	2+53	≥	13+30
Ë	2+37	û	62+85	≡	13+29
é	7+69	ù	64+85	≃	13+160
ê	62+69	ü	2+85	⊆	13+108
è	64+69	Ç	12+35	⊇	13+110

Figure 3. Composite symbols. The numbers to the right of each symbol correspond to the two simple symbols used to make the composite.

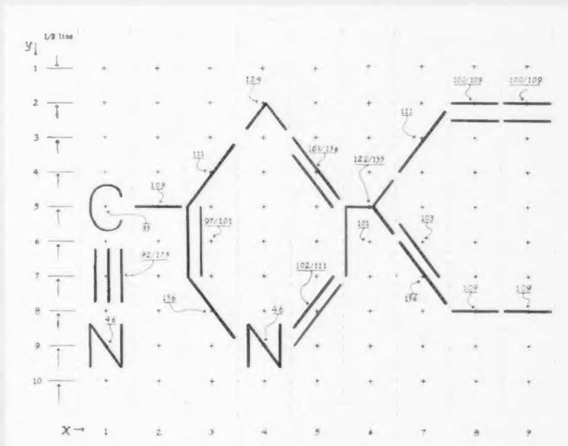


Figure 4. Detailed illustration of the use of rule segments.

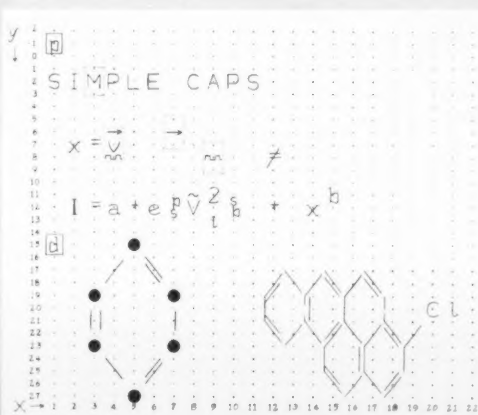


Figure 5. Model typescript page at expanded scale.

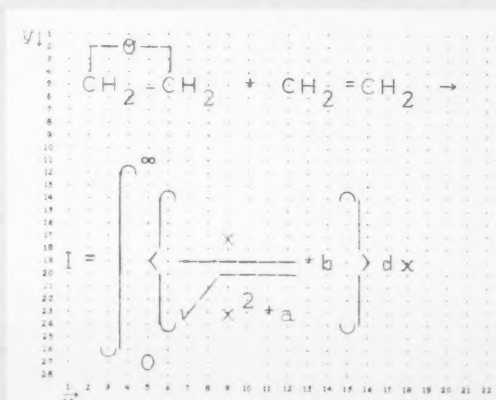


Figure 6. Two illustrative display formulas.

GPSDIC. The binary code employed in GPSDIC permits assignments of up to two, but no more than two, simple symbols to a particular x-y coordinate. Figures 4, 5, and 6 are large-scale illustrations of some of the complex notations that can be handled routinely by this system.

With GPSDIC, 244 simple symbols may be specified; however, a restriction to 188 has been established to retain adequate compatibility with USASCII. The 94 symbols in Figure 1 are the defined USASCII symbols. USASCII also defines the format effecting control code *shift out* (SO) and *shift in* (SI). These codes are provided for the purpose of causing the interchange between the defined set of symbols and a secondary set of 94. USASCII leaves the specification of such secondary sets to mutual agreement among users who wish to implement the shift-out provision. The set specified for GPSDIC is given in Figure 2.

Updating Photonuclear Data Index

Supplement No. 1 to NBS Miscellaneous Publication 277, *Photonuclear Data Index*, was published October 1, 1967. This Supplement primarily covers data published in the period from January 1965 through April 1967. In addition, it also corrects and gives the complete index entry for a number of reactions for which only partial entries were available at the time of publication of the original *Photonuclear Data Index*.

Taken together, Miscellaneous Publication 277 [1] (April 1966, 55 cents) and its Supplement [1] (October 1967, 45 cents) form a complete annotated bibliography and index to experimental data published in the field of photonuclear reactions in the period from January 1955 through April 1967. Organized by element and isotope, each entry in the index supplies quantitative information for a specific reaction on the ranges of excitation energy, source energy, detected particle energy, and emission angles for products in each reference. Information is also given on the type of measurement and detector used.

It is planned to issue supplements to the *Photonuclear Data Index* at about 20-month intervals. Future issues will contain cumulative entries. This will continue until a given supplement covers the data published over a five- or ten-year period. At that time a new cumulative supplement will be started. It is hoped that this will prevent the Index and its supplements from becoming unwieldly.

Selected Values of Chemical Thermodynamic Properties

Selected Values of Chemical Thermodynamic Properties, Tables for the First Thirty-Four Elements in the Standard Order of Arrangement, Technical Note 270–3 [1] ($1.25), contains material prepared as a revision of the tables of Series I of NBS Circular 500, *Selected Values of Chemical Thermodynamic Properties.* This Technical Note supersedes Technical Notes 270–1 and 270–2, containing all their tables, including corrections, as well as extending the number of compounds covered. Compounds of the following elements are covered: oxygen, hydrogen, helium, neon, argon, krypton, xenon, radon, fluorine, chlorine, bromine, iodine, astatine, sulfur, selenium, tellurium, polonium, nitrogen, phosphorus, arsenic, antimony, bismuth, carbon, silicon, germanium, tin, lead, boron, aluminum, gallium, indium, thallium, zinc, and cadmium. The tables contain values of the enthalpy and Gibbs energy of formation; enthalpy, entropy, and heat capacity at 298.15 K (25 °C); and the enthalpy of formation at 0 K, for all inorganic substances and organic molecules having not more than two carbon atoms.

As additional revised tables are completed, they will be published in the Technical Note 270 Series because of the urgent need for the data. After all the tables have been revised, they will be combined with a description of the evaluation process and a listing of the sources of the data into a single publication which will be part of the NSRDS Series of critically evaluated compilations.

The authors of Technical Note 270–3 are D. D. Wagman, W. H. Evans, V. B. Parker, I. Halow, S. M. Bailey, and R. H. Schumm.

International Cooperation in Standard Reference Data Projects

Individual standard reference data activities are leading to an increasing number of international projects. Several projects begun unilaterally have become recognized as bases for international cooperation. An example is the excellent reception which the recent Madrid Conference on Molecular Spectroscopy gave to the recommendations presented by Norman Jones and Richard Lord on applications of computers to digitization, storage, and retrieval of spectral data. The presentation was derived from conclusions reached previously at the *ad hoc* Conference on Computer Utilization in Spectroscopy, organized for the Office of Standard Reference Data by the National Academy of Sciences-National Research Council. These recommendations provided the basis for a voluntary set of contributions and eventual exchange of high-quality infrared spectra. The criteria for the evaluation of infrared spectra developed by the Board of Managers of the Coblentz Society for the Office of Standard Reference Data provide a tentative definition of quality standards to be observed. A second international project arises out of the *Tables of Bimolecular Gas Phase Kinetic Data* which Aubrey F. Trotman-Dickenson has prepared under an NBS contract. Professor Trotman-Dickenson's future updatings of this work will be sponsored by the British Office of Scientific and Technical Information.

[1] Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for the price indicated.

agreement with the UT2 scale by making step adjustments of 100 ms as necessary. Each pulse indicates that the earth has rotated approximately 15 arcseconds about its axis since the previous one. Adjustments are made at 0000 UT on the first day of a month. *There will be no adjustment made on May 1, 1968.* The pulses occur at intervals that are longer than one second by 300 parts in 10^{10} due to an offset in carrier frequency coordinated by the Bureau International de l'Heure (BIH), Paris, France.

Radio station WWVB (Fort Collins, Colo.) broadcasts seconds pulses derived from the NBS Time Standard (NBS–III) with no offset. Step adjustments of 200 ms are made at 0000 UT on the first day of a month when necessary. BIH announces when such adjustments should be made in the scale to maintain the seconds pulses within about 100 ms of UT2. *There will be an adjustment made on May 1, 1968. The seconds pulses emitted from WWVB will be retarded 200 ms.*

[1] **Impedance measurements in coaxial waveguide systems,** by R. L. Jesch and R. M. Jickling, Proc. IEEE **55**, 912–923 (1967).

[2] **Standardization of precision coaxial connectors,** by B. O. Weinschel, Proc. IEEE **55**, 923–930 (1967).

[3] Unpublished paper.

[4] In performing microwave calibrations, a considerable amount of time usually is needed to prepare the system for operation. Much of this preparation is related to adjustment of the system to the frequency selected for the calibration. Time and cost often can be reduced by minimizing the number of times the operating frequency of the calibration system must be readjusted.

[5] **Bolometric microwave power calibration techniques at the National Bureau of Standards,** by R. F. Desch and R. E. Larson, IEEE Trans. Instr. Meas. **IM–12**, No. 1, 29–33 (1963).

[6] **Determination of efficiency of microwave bolometer mounts from impedance data,** by D. M. Kerns, J. Res. NBS **42**, 579–585 (1949).

[7] **An improved method of measuring efficiencies of ultrahigh-frequency and microwave bolometer mounts,** by R. W. Beatty and F. Reggia, J. Res. NBS **54**, 321–327 (1955).

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[9] **Experimental confirmation of barretter substitution error,** by J. W. Adams and R. F. Desch, IEEE Trans. MTT, to be published.

[10] **Improved calibration accuracy for microwave bolometric devices,** NBS Tech. News Bull. **51**, No. 8, 174 (1967).

NBS TO HOST TWO CONFERENCES IN MAY

ACM Technical Symposium

The Bureau is joining with the Washington, D.C., chapter of the Association for Computing Machinery in sponsoring the 7th Annual Technical Symposium, having as its theme "The Technology Gap." The sessions will be held May 16, 1968, at the Bureau's Gaithersburg (Md.) facilities under the chairmanship of G. Richard Reed (of the Naval Command Systems Support Activity). Program cochairmen are Kenneth Joseph (National Aeronautics and Space Administration) and Richard Coles (Orchard-Hays & Co., Inc.).

The Symposium will open with a keynote address by H. R. J. Grosch, Director of the Bureau's Center for Computer Sciences and Technology. The first session will consist of a panel discussion of the present technology gap. Following this, the attendees will be offered choices among concurrent sessions devoted to such areas as: the use of on-line computers, the social implications of computers, program documentation, standards applicable to instruction in computer techniques, and numerical analysis.

Members of the ACM are urged to attend, as are others interested in the Symposium's content. Registration begins at 8:30 a.m. on Thursday, May 16. Registration fees are $7 for Washington chapter members and $9 for all others (includes luncheon). Advance registrations are being accepted by Miss Sharon Rose, Registration Chairman, ACM Symposium, National Bureau of Standards, Washington, D.C. 20234.

Material Failures Conference

On May 1, 1968, an all-day conference on material failures will be held at the NBS Gaithersburg (Md.) laboratories. This program, sponsored by the Washington Chapters of the American Society for Metals and the American Society of Mechanical Engineers, and by the National Bureau of Standards, will present an interdisciplinary look at the failure of materials in service.

Six nationally known authorities in mechanical engineering, metallurgy, and nondestructive testing will present their views. Ralph Nader, well-known critic of automotive safety, will also address the conference.

Registration may be made in advance by mailing a check for $8 ($10 after April 21), payable to MATERIAL FAILURES CONFERENCE, to:

Dr. H. P. Utech
Rm. B164, Materials Bldg.
National Bureau of Standards
Washington, D.C. 20234

PUBLICATIONS of the National Bureau of Standards*

PERIODICALS

Technical News Bulletin, Volume **52,** No. 3, March 1968. 15 cents. Annual subscription: Domestic, $1.50; foreign, $2.25. Available on a 1-, 2-, or 3-year subscription basis.

Journal of Research of the National Bureau of Standards

Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic, $5; foreign, $6. Single copy, $1.

Section B. Mathematical Sciences. (Formerly *Mathematics and Mathematical Physics.*) Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75. Single copy, 75 cents.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, $2.75; foreign, $3.50. Single copy, 75 cents.

CURRENT ISSUES OF THE JOURNAL OF RESEARCH

J. Res. NBS **72A** (*Phys. and Chem.*), No. 2 (Mar.–Apr. 1968), $1.

Constant pressure flame calorimetry with fluorine II. The heat of formation of oxygen difluoride. R. C. King and G. T. Armstrong.

The heat of formation of boron carbide. E. S. Domalski and G. T. Armstrong.

Solubility of tris(hydroxymethyl)aminomethane in water-methanol solvent mixtures and medium effects in the dissociation of the protonated base. P. Schindler, R. A. Robinson, and R. G. Bates.

Calibration of the nickel dimethylglyoxime spectral shift at pressures to 20 kilobars for use in spectroscopic measurement. H. W. Davis.

Second virial coefficient of He^4 in the temperature range from 2 to 20 °K. M. E. Boyd, S. Y. Larsen, and H. Plumb.

Diffusion rates in organic nuclear materials. A. L. Dragoo.

Effect of oxide additions on the polymorphism of tantalum pentoxide. (System Ta_2O_5–TiO_2). J. L. Waring and R. S. Roth.

Effect of statistical counting errors on wavelength criteria for x-ray spectra. J. S. Thomsen and F. Y. Yap.

Thermodynamic properties of ammonia as an ideal gas. L. Haar.

J. Res. NBS **72B** (*Math. Sci.*), No. 1 (Jan.–Mar. 1968), 75 cents.

Means and the minimization of errors. M. Aissen.

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Principal submatrices III: Linear inequalities. R. C. Thompson.

The diophantine equation $P(x,y) = (xy + d)z$. C. F. Osgood.

A note on the G-transformation. H. L. Gray and T. A. Atchison.

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Solutions of the time-dependent Klein-Gordon and Dirac equations for a uniform electric field. V. W. Myers.

Analysis of a market split model. J. M. McLynn, A. J. Goldman, P. R. Meyers, and R. H. Watkins.

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Analytical coordination chemistry: Titrimetry, gravimetry, flame photometry, spectrophotometry, and gas evolution, July 1966 to June 1967, Ed. O. Menis, Tech. Note 424 (Jan. 1968), 55 cents.

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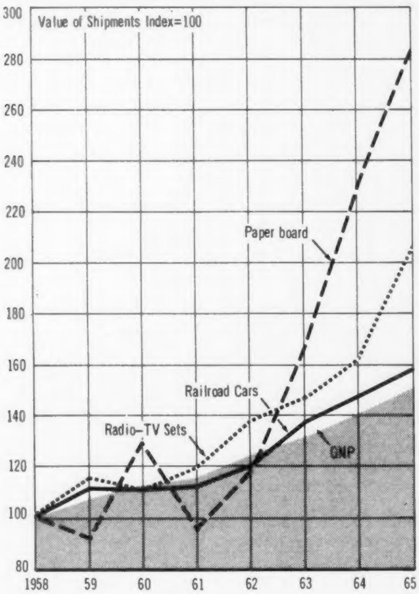

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